SQUIRE SANDERS | LEGAL COUNSEL WORLDWIDE

SQUIRE, SANDERS & DEMPSEY L.L.P.
4900 Key Tower
127 Public Square
Cleveland, Ohio 44114-1304
Office: +1.216.479.8500
Fax: +1.216.479.8780
Direct Dial: +1.216.479.8332
dmcwilliams@ssd.com

May 6, 2003

VIA OVERNIGHT DELIVERY

Gwendolyn Massenburg
Remedial Project Manager
U.S. EPA, Region V (SR-6J)
77 West Jackson Blvd.
Chicago, IL 60604-3590

Re: CRS Site Quality Assurance Project Plan Re-Submission

Dear Ms. Massenburg:

On behalf of the CRS Site Group, I am submitting with this letter the revised pages of the CRS Site Quality Assurance Project Plan (QAPP). These revisions are the result of discussions with Ms. Roberman, U.S. EPA QAPP Reviewer, and Ms. Massenburg, U.S. EPA Remedial Project Manager. We understand that these changes resolve all remaining obstacles to U.S. EPA approval of the QAPP for the CRS Site.

The location and purpose of the revisions are summarized below:

- A revised *Table of Contents* has been included.
- Section 3.2 *Sampling Program* (including subsections 3.2.1 through 3.2.4) was revised slightly based on comments from the USEPA on the Work Plan and Field Sampling Plan – this section agrees with the current version of the Work Plan (Revision I, April 2003) and Field Sampling Plan (Revision I, April 2003).
- Table 4-2 *Chemicals of Concern* was revised (a copy was faxed to Ms. Roberman on 22 April 2003) based on conversations with Ms. Roberman and Ms. Massenburg.
- Section 7.6 *Rationale For Sample Design* – was revised slightly based on changes to the Work Plan (Revision I, April 2003) and Field Sampling Plan (Revision I, April 2003).
- Table 7-1 *Analytical Methods and Total Number of Analysis* – was revised based on the change of metal analysis methods, as a result of changes to Table 4-2 and discussion with Ms. Roberman.

- Various subsections of Section 8.1 *Sampling Procedures and Methods* (8.1.1, 8.1.2, 8.1.3, 8.1.6, and 8.1.7) were revised slightly based on revisions to the Work Plan (Revision I, April 2003) and Field Sampling Plan (Revision I, April 2003).
- Section 8.5 Investigative Derived Waste Management was revised slightly based on revisions to the Work Plan (Revision I, April 2003) and Field Sampling Plan (Revision I, April 2003).

Please insert these revised pages into the previously submitted QAPP and remove the pages replaced. We understand that this updated QAPP is now complete. Please confirm that no further revisions are necessary for the CRS Site QAPP. If you have any questions or concerns, please contact me directly.

Sincerely,

Douglas McWilliams
as per TBS

Douglas McWilliams
CRS Site Group Chairperson

Enclosures

Copy: Thomas Nash, Associate Regional Counsel, U.S. EPA
Lawrence Antonelli, Ohio EPA
Gary Gifford, CRS Technical Committee Chairperson

QUALITY ASSURANCE PROJECT PLAN

FOR THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY AT CHEMICAL RECOVERY SYSTEMS, INC. ELYRIA, OHIO

REVISION II

FEBRUARY 2003

Prepared for: The CRS Site Group

Prepared by: PARSONS

Parsons Project Manager, Peter Gelman **Date**

Project QA Manager and Chemist, Diane Short **Date**

Laboratory QA/QC Manager, Beth Lambert **Date**

CRS Site Group Project Coordinator, Douglas McWilliams **Date**

U.S. EPA Region 5 Remedial Project Manager, Gwendolyn Massenburg **Date**

U.S. EPA Region 5 Quality Assurance Reviewer **Date**

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3.0 PROJECT TASK / DESCRIPTION

The purpose of this Section is an overview of the work. It describes the approach taken to address the project's objectives and the problem defined in Section 2.0, connecting what is needed to how it will be obtained.

3.1 INTRODUCTION

Existing information on chemicals of concern, potential source areas, direct discharges, potential release mechanisms, affected media, known and potential routes of migration were used to develop a conceptual site model (CSM) for the CRS site (Figure 2-1). Based on review and evaluation of the existing information from previous sampling events (Figure 3-2), data gaps have been defined. The primary goal of the RI/FS process, at this site, is to fill these data gaps with the results of the site investigation activities, which will determine the extent of chemicals of concern in the site media, and to use these data to determine remedial actions, if necessary.

Sample matrices for the scope of this QAPP include: soil, groundwater, surface water, and sediment.

3.2 SAMPLING PROGRAM

The Sampling Program (including Standard Operating Procedures (SOPs), cleaning and decontamination of equipment/sample containers, field equipment maintenance, testing, acceptance, and inspection requirements, for supplies/sample containers) is outlined in detail in the text of the FSP. Details of sample handling, sample custody, chain-of-custody, and field custody procedures are also outline in the FSP.

The planned boring locations, discussed below, are shown in Figure 3-1 (of this document) and summarized in Table 5 in Section 5.0 of the Work Plan.

During the RI, five soil borings will be installed in each area of concern. Borings will be placed to the north, south, east, west, and in the center of each area of concern to determine the horizontal extent of impacted soil. The boring advanced in the center of each AOC will be completed as a one-inch temporary monitoring well. In addition to the one-inch temporary monitoring wells, eight two-inch permanent wells will be installed at the site. Three permanent wells will be deep (40 to 50 feet bsg) and five will be shallow (15 to 25 feet bsg) in depth to determine the vertical extent of groundwater impact. A total of 19

groundwater samples will be collected at the site (8 temporary monitoring wells, 8 permanent monitoring wells, MW-2, and L-2 and L-3 located across Locust Street). Five surface water and sediment samples will be collected upgradient and adjacent to the site property. In addition, 4 geotechnical soil samples will be collected at the site to determine the sand size and screen slot size during monitoring well construction. Geotechnical samples will also be utilized in site characterization.

Sampling activities will occur in three phases at the site. The first phase will include the installation of 39 soil borings (including temporary monitoring wells), using a Geoprobe™ drill rig. During this time, soil samples will be collected from all soil borings and submitted for laboratory analysis. Groundwater samples will also be collected from the temporary monitoring wells and submitted for laboratory analysis. After all soil borings are completed, four geotechnical soil samples (two in fill material and two in natural soil) will be collected. The locations of the geotechnical samples will be based on field observations during soil borings installation. All soil borings will be backfilled with bentonite holeplug, after all soil samples are collected. The temporary monitoring wells will be completed with a temporary well pad and will be capped. These wells will be utilized as "piezometers" to aid in the installation of the permanent monitoring wells; however, the temporary monitoring wells will be sampled only once during the first phase of sampling.

After receipt of preliminary sample results from the first phase of sampling, the second phase of sampling will occur at the site. The second phase of sampling will include the installation of the two-inch permanent wells, soil sampling, groundwater sampling (of the newly installed permanent wells, and existing wells (MW-2, L-2, and L-3)), sediment and surface water sampling. The location of the permanent monitoring wells may be adjusted, based on the results of the first phase of sampling (soil and groundwater samples). At this time, the sediment and surface water samples will be collected and submitted for laboratory analysis. If it is determined that PCBs are present in the site soil and / or groundwater, surface water and sediment samples may be analyzed for PCBs. The temporary monitoring wells will be removed and backfilled with bentonite, prior to the completion of field activities.

After the final data from the first and second phases of sampling is received from the laboratory, it will be validated and evaluated. Based on the results of the field sampling, additional data may be collected to determine extent (if needed), to aid in feasibility studies (if needed), and/or to characterize IDW (if needed).

All samples will be located in the field using an aerial photograph with a known scale and existing site structures. All sampling locations will be marked with a surveyors stake marked with the sampling location numbers, and professionally surveyed.

3.2.1 Soil Sampling

The field sampling at the CRS site will occur in phases. During the first phase, 39 soil borings will be advanced and sampled (eight of which will be completed as one-inch temporary monitoring wells), as a part of the soil sampling program to evaluate the potential areas of concern. Five soil borings will be placed in each AOC (the center, the north, south, east, and west) to define the extent and magnitude of chemicals of concern in the site soil (Figure 3-1). The soil boring, located in the center of each area of concern, will be completed as a one-inch temporary monitoring well. These temporary monitoring wells will be utilized to All soil samples submitted for laboratory analysis will be analyzed for VOCs, SVOCs, PCBs, and TAL metals (Table 7-1). determine the groundwater quality in each AOC. The soil borings will be advanced by direct-push technologies (e.g. Geoprobe™) to approximate depths of 15 feet below grade, top of the groundwater interface, or bedrock (whichever is achieved first). Soil samples will be collected continuously from the surface grade to the bottom of the boring in two-foot intervals. All soil samples will be field screened for VOCs, using a PID. A maximum of three grab soil samples from each boring will be submitted to the laboratory for analysis: a surface soil sample with the highest PID reading from the depth interval of 0 to 3 feet bgs; a sample from the depth interval just above the groundwater interface; and the subsurface sample collected at the depth interval with the highest PID reading above the groundwater interface. If the sample from the depth interval just above the groundwater interface is also the sample with the highest PID reading, only two soil samples will be submitted for laboratory analysis from that boring. All soil samples submitted for laboratory analysis will be analyzed for VOCs, SVOCs, PCBs, and TAL metals (Table 7-1).

If overt signs of impact are noticed during boring installation (e.g. staining, odors, etc.), boring will be abandoned and the Geoprobe™ will step out in the opposite direction of the area of concern to determine the extent of the impact. All borings will be abandoned upon collecting soil samples. Boring will be abandoned by filling the boring with bentonite holeplug.

Boring locations will be determined in the field using high-resolution aerial photographs with a known scale. Based on the aerial photograph, boring locations can be scaled off using permanent onsite structures (buildings, foundations, monitoring wells, railroad tracks, etc.). During the site investigation, a stake with the boring identification number will be placed at all boring locations (including temporary monitoring wells). After all field activities are completed, the boring locations, temporary monitoring wells, and permanent monitoring wells will be professionally surveyed.

Geotechnical samples will be collected (2 in natural soils and 2 in fill materials) after all soil borings have been completed to aid in the geotechnical sampling location placement. Geotechnical samples will be analyzed for grain size, moisture content, and atterberg limits. Results will be utilized in determining the screen slot size and sand pack to be used in monitoring well construction. In addition, geotechnical information will be utilized in site characterization.

3.2.2 Monitoring Well Installation and Groundwater Sampling

During the second phase of sampling activities, eight two-inch permanent monitoring wells will be installed at the site, five of which will be shallow (15 to 25 feet bgs) and three will be deep (40 to 50 feet bgs) to define the horizontal extent and magnitude of chemicals of concern in site groundwater. The shallow wells will be installed to monitor the uppermost saturated zone at the site, near potential source areas. The three deeper bedrock wells will be installed to evaluate the vertical extent of impacted groundwater at the site. These data will augment the data collected from the eight temporary monitoring wells, which will be installed and sampled at the site during the first phase of sampling. The locations of the permanent monitoring wells may be adjusted based on the preliminary results of the first phase of sampling activities. Soil borings, to be completed as permanent monitoring wells, will be advanced using a drill rig equipped with hollow stem augers. Soil samples will be

collected continuously from the surface to the bottom of the boring in two-foot intervals. All samples will be field screened for VOCs, using a PID. A maximum of two grab soil samples will be submitted from each monitoring well location for laboratory analysis: the sample from the depth interval just above the groundwater interface and the subsurface sample with the highest PID reading above the groundwater interface. If the sample from the depth interval just above the groundwater interface is also the sample with the highest PID reading, only one soil sample from that boring will be submitted for laboratory analysis.

Each of newly installed permanent and temporary wells (16 total), along with the existing on-site well (MW-2) and two existing wells along Locust Street (previously installed by Engelhard Corporation), will be appropriately sampled for VOCs, SVOCs, PCBs, TAL metals, chloride, methane, ethane, and ethene (Table 7-1). All wells will be allowed one week after the completion of well installation activities to equilibrate, prior to sampling. MNA parameters (dissolved oxygen, pH, conductivity, temperature, oxidation-reduction potential (ORP), sulfate, nitrate, and ferrous iron) will also be collected at each monitoring well location to determine if natural attenuation processes are occurring on dissolved organic compounds present in on-site groundwater. The one-inch temporary monitoring wells will be sampled only during the first phase of sampling.

Monitoring well sample locations will be placed in the field using aerial photographs. Following site investigation activities, monitoring wells will be professionally surveyed.

3.2.3 Surface Water Sampling

To determine if surface water is impacted five surface water samples (including one to be collected directly from the storm sewer outfall pipe effluent that drains Locust Street and adjacent industrial facilities) will be collected and analyzed for VOCs, SVOCs, and TAL metals. Samples will be analyzed as detailed in Table 7-1. The farthest downstream sample will be collected first, working to the farthest upstream sample to minimize suspended sediments from upstream impacting the downstream samples. In addition, the storm sewer will be surveyed with a camera to determine if any areas of the pipe have been damaged.

3.2.4 Sediment Sampling

To determine if stream sediment has been impacted by former site operations, five sediment samples will be collected from identical locations as the surface water samples and

analyzed for VOCs, SVOCs and metals. Samples will be analyzed as detailed in Table 7-1. Surface water samples will be collected prior to the collection of the sediment samples. In addition, the farthest downstream sample will be collected first, working to the farthest upstream sample to minimize suspended sediments from upstream impacting the downstream samples.

3.3 PRODUCTS TO BE PRODUCED

Two technical reports will be prepared as part of the RI/FS. The format for each report is detailed below.

3.3.1 Remedial Investigation Report

The Remedial Investigation (RI) Report presents the site history and environmental setting, describes the project activities including methods and QA/QC, and presents and evaluates field and analytical laboratory data. The data evaluation includes the assessment of chemical migration and fate and the potential risk to human health and the environment.

3.3.2 Feasibility Study

The Feasibility Study (FS) Report will describe and justify the development of a preferred remedial alternative.

3.3.3 Decision Documents

Decision documents summarize the rationale for selecting a particular remedial action, including the No Action Alternative, at the site. The decision document is the official selection of a remedial alternative and requires the formal concurrence of all appropriate agencies.

3.4 PROJECT SCHEDULE

The schedule for sampling and analysis is attached at the end of this Section (Figure 3-2). The schedule will be updated periodically as the Work progresses.

Figure 3-1 Proposed Sampling Locations

detail in the following section for each media being investigated (soil, groundwater, surface water, and sediment) and summarized in Table 4-1.

4.1 DEFINE EXPOSURE SCENARIOS

Based on the existing data and the CSM, the following exposure pathways have been defined as: soil to inhalation; soil ingestion; soil dermal exposure; soil to indoor air – inhalation; groundwater to indoor air – inhalation; surface water direct contact; stream sediment direct contact; groundwater exposure (industrial use) onsite and offsite; and stream sediment (aquatic animals). However, it was determined that groundwater and surface water ingestion, and surface water to groundwater pathways are not considered complete. Future land use is expected to be industrial, which may be reinforced with deed restrictions.

Based on historical data, potential chemicals of concern (COCs) have been identified and are summarized in Table 4-2. Project Action Levels have been developed and are summarized in Table 4-2. It should be noted that the project action levels may change and ARARs may be developed as more current analytical data becomes available.

4.2 IDENTIFY THE DECISION

Based on historical data, the following questions for the site have been developed:

- Do concentrations of chemicals of concern, in the site soils, groundwater, surface water and stream sediments, exceed regulatory/risk limits?
- What risk to human health or the environment and what exposure pathways exist for the chemicals of concern in the site soils, groundwater, surface water or stream sediment?

Based on the above questions, alternative actions have been identified:

- Recommend that the site requires further assessment or possible response action.

Based on these identified alternative actions the following Decision Statement has been developed:

- Determine whether site impact poses an unacceptable risk to human health or the environment and requires further consideration or a response action, or recommend that no further action is needed.

Table 4-2
Chemicals of Concern in Soil and Sediment Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/kg)	Project Quantitation Limits (ug/kg)		Analytical Method Detection Limit (MDL) (ug/kg)		Achievable Laboratory MDL (ug/kg)	
			Sodium Bisulfate	Methanol	Sodium Bisulfate	Methanol	Sodium Bisulfate	Methanol
VOCs Method SW 846 5035								
benzene	71-43-2	1,300	5.00	40	0.096	6.90	5.00	40
1,2-dichloroethane	107-06-2	600	5.00	40	0.2	13	5.00	40
1,2-dichloropropane	78-87-5	740	5.00	40	0.17	9.4	5.00	40
carbon tetrachloride	56-23-5	550	5.00	40	0.15	14	5.00	40
ethylbenzene	100-41-4	20,000	5.00	40	0.10	6.50	5.00	40
1,1,2,2-tetrachloroethane	79-34-5	930	5.00	80	0.18	10	5.00	80
tetrachloroethene	127-18-4	3,400	5.00	40	0.10	11	5.00	40
toluene	108-88-3	520,000	5.00	80	0.18	5.60	5.00	80
trichloroethene	79-01-6	110	5.00	40	0.10	8.70	5.00	40

A Low Level Scan (using sodium bisulfate as a preservative) will be utilized during the site investigation. A High Level Scan (methanol preservation) may be utilized if the soil sample reacts with the sodium bisulfate preservative.

Table 4-2 (Continued)
Chemicals of Concern in Soil and Sediment Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/kg)	Project Quantitation Limit (ug/kg)	Analytical MDL (ug/kg)	Achievable Laboratory MDL (ug/kg)
SVOCs Method SW 846 8270					
acenaphthylene	208-96-8	330	330	19	330
benzo (a) anthracene	56-55-3	2,100	330	23	330
benzo (b) fluoranthene	205-99-2	2,100	330	20	330
benzo (ghi) perylene	191-24-2	330	330	16	330
benzo (a) pyrene	50-32-8	210	210	15	210
bis (2-chloroethyl) ether	111-44-4	550	330	18	330
dibenz (a,h) anthracene	53-70-3	210	210	18	210
hexachlorobenzene	118-74-1	1,100	330	22	330
indeno (1,2,3-cd) pyrene	193-39-5	2,100	330	19	330
2-methylnaphthalene	91-57-6	330	330	17	330
n-nitroso-di-n-dipropylamine	621-64-7	250	250	15	250
phenanthrene	65996-93-2	330	330	21	330
PCBs Method SW 846 8082					
aroclor 1254	11097-69-1	740	330	4.7	264
aroclor 1232	11141-16-5	740	330	8.2	264
TAL Metals Method SW 846 6020/6010/7470					
arsenic	7440-38-2	1,600	1,000	280	1,000
cadmium	7440-43-9	450,000	1,000	16	500
lead	7439-92-1	750,000	1,000	190	1,000

Table 4-2 (Continued)
Chemicals of Concern in Surface Water and Groundwater Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/L)	Project Quantitation Limit (ug/L)	Analytical MDL (ug/L)	Achievable Laboratory MDL (ug/L)
SVOCs Method SW 846 8260					
acetone	67-64-1	610	10	1.10	10
benzene	71-43-2	1.00	1.00	0.37	1.00
bromodichloromethane	75-27-4	1.00	1.00	0.32	1.00
bromoform	75-25-2	8.50	1.00	0.37	1.00
bromomethane	74-83-9	8.70	1.00	0.30	1.00
carbon disulfide	75-15-0	1,000	1.00	0.24	1.00
carbon tetrachloride	56-23-5	1.00	1.00	0.37	1.00
chlorobenzene	108-90-7	110	1.00	0.38	1.00
chloroethane	151-67-7	4.60	1.00	0.29	1.00
chloroform	67-66-3	6.20	1.00	0.35	1.00
chloromethane	74-87-3	1.50	1.00	0.49	1.00
dibromochloromethane	124-48-1	1.00	1.00	0.37	1.00
cis-1,3-dichloropropene	10061-01-5	1.00	1.00	0.35	1.00
1,1-dichloroethane	75-34-3	810	1.00	0.30	1.00
1,2-dichloroethane	107-06-02	1.00	1.00	0.28	1.00
1,1-dichloroethene	75-35-4	340	1.00	0.31	1.00
1,2-dichloroethene (total)	540-59-0	61	1.00	0.66	1.00

Table 4-2 (Continued)
Chemicals of Concern in Surface Water and Groundwater Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/L)	Project Quantitation Limit (ug/L)	Analytical MDL (ug/L)	Achievable Laboratory MDL (ug/L)
SVOCs Method SW 846 8260					
1,2-dichloropropane	78-87-5	1.00	1.00	0.41	1.00
ethylbenzene	100-41-4	2.90	1.00	0.41	1.00
methylene chloride	75-09-2	4.30	1.00	0.29	1.00
styrene	100-42-5	1,600	1.00	0.35	1.00
tetrachloroethene	79-01-6	0.66	1.00	0.42	1.00
toluene	108-88-3	720	1.00	0.39	1.00
trans-1,2-dichloroethene	156-59-0	0.5	1.00	0.33	0.5
trans-1,3-dichloropropene	563-54-2	1.00	1.00	0.35	1.00
1,1,1-trichloroethane	71-55-6	3,200	1.00	0.39	1.00
1,1,2-trichloroethane	79-00-5	1.00	1.00	0.36	1.00
1,1,2,2-tetrachloroethane	79-34-5	1.00	1.00	0.36	1.00
trichloroethene	79-01-6	1.00	1.00	0.42	1.00
vinyl chloride	75-01-4	1.00	1.00	0.36	1.00
xylenes (total)	106-42-3	210	1.00	0.44	1.00
SVOCs Method SW 846 8270					
benzo (a) anthracene	56-55-3	10	10	0.7	10
benzo (a) pyrene	50-32-8	10	10	0.73	10
bis (2-chloroethyl) ether	111-44-4	10	10	0.97	10

Table 4-2 (Continued)
Chemicals of Concern in Surface Water and Groundwater Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/L)	Project Quantitation Limit (ug/L)	Analytical MDL (ug/L)	Achievable Laboratory MDL (ug/L)
SVOCs Method SW 846 8270					
bis (2-ethylhexyl) phthalate	117-81-7	10	10	2.7	10
carbazole	86-74-8	10	10.00	0.82	10
2-chlorophenol	95-57-8	30	10.00	1.00	10
1,2-dichlorobenzene	95-50-1	370	10.00	0.81	10
1,3-dichlorobenzene	541-73-1	10	10.00	0.83	10
1,4-dichlorobenzene	106-46-7	10	10.00	0.77	10
3,3-dichlorobenzidine	91-94-1	50	50.00	0.93	50
2,4 dimethylphenol	1300-71-6	730	10.00	0.85	10
di-n-butylphthalate	87-74-2	10	10.00	0.67	10
4,6-dintro-2-methylphenol	534-52-1	50	50.00	1.30	50
2,4-dinitrotoluene	121-14-2	73	10.00	0.79	10
2,6-dinitrotoluene	606-20-2	36	10.00	0.57	10
hexachlorobenzene	118-74-1	10	10.00	0.74	10
hexachlorobutadiene	87-68-3	10	10.00	0.73	10
hexachloroethane	67-72-1	10	10.00	0.74	10
isophorone	78-59-1	71	10	0.63	10
2-methylnaphthalene	91-57-6	10	10	0.64	10

Table 4-2 (Continued)
Chemicals of Concern in Surface Water and Groundwater Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/L)	Project Quantitation Limit (ug/L)	Analytical MDL (ug/L)	Achievable Laboratory MDL (ug/L)
SVOCs Method SW 846 8270					
2-methylphenol	95-48-7	1,800	10	1.10	10
naphthalene	91-20-3	10	10	0.75	10
2-nitroaniline	88-74-4	10	50	0.70	50
3-nitroaniline	99-09-2	50	50	0.71	50
n-nitroso-di-n-dipropylamine	621-64-7	10	10	0.67	10
pentachlorophenol	87-86-5	10	10	2.40	10
2,4,6-trichlorophenol	933-75-5	10	10	1.70	10
PCBs Method SW 846 8082					
aroclor-1254	11097-69-1	0.034	0.20	0.00201	0.01
aroclor-1248	1336-36-3	0.034	0.20	0.00156	0.01
TAL Metals Method SW 846 6020/6010/7470					
aluminum	7429-90-5	36,000	200	16	50
antimony	7440-36-0	15	100	0.12	2.00
arsenic	7440-38-2	5	300	0.21	5.00
barium	7440-39-3	2,600	200	0.057	1.00
cadmium	7440-43-9	18	10	0.075	1.00
chromium	7440-47-3	110	10	0.16	2.00

Table 4-2 (Continued)
Chemicals of Concern in Surface Water and Groundwater Samples
CRS Site
Elyria, Ohio

Analyte	CAS Number	Project Action Limits (ug/L)	Project Quantitation Limit (ug/L)	Analytical MDL (ug/L)	Achievable Laboratory MDL (ug/L)
TAL Metals Method SW 846 6020/6010/7470					
copper	7440-50-8	1,500	100	0.33	2.00
iron	7439-89-6	11,000	100	17	20
manganese	7439-96-5	880	100	0.20	1.00
nickel	7440-02-0	730	100	0.059	2.00
thallium	7440-28-0	2.40	2,000	0.033	1.00
vanadium	7440-62-2	260	100	0.34	5.00
zinc	1314-13-2	11,000	100	2.20	10

metals were detected in the sediment sample collected adjacent to the Locust Street storm sewer outfall. Specific analysis to be performed, for each media, are outlined in Tables 7-1. A list of the analytical parameters to be analyzed and their method detection limits, for the site, are located in Table 4-2.

7.6 RATIONALE FOR THE SAMPLE DESIGN

Based on the above data gaps and DQOs, a sampling plan has been developed to obtain information on the presence, extent, and magnitude of COCs in the site's soil, groundwater, surface water, and sediment. Sampling activities will target areas of concern, as outlined in Table 4-1 of the Work Plan. The sampling activities have been developed to define the extent of impact (both horizontally and vertically) in the site soil and groundwater, which will satisfy the data needs of the site. The details of the sampling plan are outlined in Section 8.0.

Based on USEPA guidance, information on the contaminant source(s) and environmental setting should be determined prior to developing a sampling program. Useful data may be found in the following sources, when available: facility assessment reports, facility records and files, regulatory agencies, design and construction diagrams, permits, environmental studies, interviews with facility personnel, environmental audit reports, environmental insurance policies, etc. (USEPA, 1989).

A review of data from a variety of sources has revealed that a substantial amount of information is available regarding historic site operations, including aerial photographs from 1966 and 1978, during solvent recovery operations at the site. Data regarding areas of likely impact are also available from the investigation and remedial actions, performed in the early 1980s, and the state and federal site assessments conducted in the mid 1990s (see Section 2.4). This information has been used to identify areas of concern for targeted sampling to maximize the efficiency of the remedial investigation. Areas of concern have been divided into five categories: Former Drum Storage Areas, Former Tanker Staging Areas, the Still Buildings (the Former Brighton Still Building and AST area and the Rodney Hunt Still Building), and the Storm Sewer Outfall (Figure 2-3).

Targeting known areas of impact (judgmental sampling) is appropriate, under USEPA guidance, when specific information exists on the potential configuration of a release or areas of impact (USEPA, 1989). Many releases are likely to fall into this category, because the site layout or characteristics will often indicate areas of potential impact. Judgmental sampling will generally bias the data toward higher contaminant concentrations. In many cases, this approach will suit the needs of the remedial field investigation (USEPA, 1989).

Table 7-1
Analytical Methods and Total Number of Analyses
CRS Site
Elyria, Ohio

Parameter	Matrix	Analytical Method	Number of Analyses	Trip Blanks	Equipment Blanks	Duplicates	MS/ MSD	Total Analyses
Soil – (Geoprobe borings and monitoring well installation)								
VOCs	Soil	5035	133	7	7	--	6	153
SVOCs	Soil	8270	133	--	--	--	6	139
Metals (TAL List)	Soil	6010/6020/ 7470	133	--	--	--	6	139
PCBs	Soil	8082	133	--	--	--	6	139
Total Organic Carbon	Soil	415.1	4	--	--	--	--	4
Geotechnical	Soil	Various	4	--	--	--	--	4
Groundwater								
VOCs	Groundwater	8260	19	4	--	2	1	26
SVOCs	Groundwater	8270	19	--	--	2	1	22
Metals (filtered/unfiltered)	Groundwater	6010/6020/ 7470	38	--	--	2	1	41
PCBs (filtered/unfiltered)	Groundwater	8082	38	--	--	2	1	41
Chloride	Groundwater	300.0A	19	--	--	2	1	22
Methane, Ethene, and Ethane	Groundwater	RSK-175	19	--	--	2	1	22
Surface Water Samples								
VOCs	Surface Water	8260	5	1	--	1	--	7
SVOCs	Surface Water	8270	5	--	--	1	--	6
Metals	Surface Water	6010/6020/ 7470	5	--	--	1	--	6

Table 7-1 (Continued)
Analytical Methods and Total Number of Analyses
CRS Site
Elyria, Ohio

Parameter	Matrix	Analytical Method	Number of Analyses	Trip Blanks	Equipment Blanks	Duplicates	MS/ MSD	Total Analyses
Sediment Samples								
VOCs	Sediment	5035	5	1	--	1	--	7
SVOCs	Sediment	8270	5	--	--	1	--	6
Metals	Sediment	6020/6010/ 7470	5	--	--	1	--	6

- Thirty-nine soil boring (8 of which will be completed as one-inch temporary monitoring wells) will be advanced at the site. A maximum of three soil samples will be collected from these soil borings: a sample with the highest PID from 0 to 3 feet bsg; the sample from the depth interval above the groundwater interface; and the sample from the depth interval with the highest PID reading above the groundwater interface. If the sample with the highest PID reading is also the sample from the depth interval just above the groundwater interface, only two soil samples will be submitted for laboratory analysis.

- Eight soil borings to be completed, as two-inch permanent monitoring wells will be advanced at the site. A maximum of two soil samples will be collected from each of these soil borings: the sample from the depth interval above the groundwater interface and the sample from the depth interval with the highest PID reading above the groundwater interface. If the sample with the highest PID reading is also the sample from the depth interval just above the groundwater interface, only one soil sample will be submitted for laboratory analysis.

- A total of 19 groundwater samples will be collected from the site (8 temporary and 8 permanent monitoring wells, existing monitoring well MW-2, and existing wells L-2 and L-3 located across Locust Street. In addition to the proposed analytical parameters, field measurements of dissolved oxygen, oxidation-reduction potential, turbidity, pH, temperature, conductivity, nitrate, sulfate, and ferrous iron will be collected.

- Five surface water and sediment samples will be collected adjacent to and upgradient of the site, including one sample to be collected from the storm sewer outfall. The farthest downgradient sample will be collected first, working to the farthest upstream sample to minimize suspended sediments from upstream impacting the downstream samples. In addition, the surface water samples will be collected prior to the sediment samples.

- Geotechnical samples will be analyzed for Vertical Hydraulic Conductivity (ASTM-D5084), Bulk Density (D2937), Moisture Content (ASTM D2216), Grain Size (ASTM D422), and Porosity.

8.0 SAMPLING METHODS

The purpose of this Section is to detail how samples or information will be collected consistently between locations and by all sampling teams, with no contamination being introduced during collection. If a portion of the data collection will be performed without the collection of discrete samples, as in-situ or remote sensing monitoring, the information in this element details how the instruments will be deployed and operated to avoid contamination and to ensure maintenance of the proper data.

8.1 SAMPLING PROCEDURES AND METHODS

During this investigation soil, groundwater, surface water, and sediment samples will be collected. In addition, field analysis will be performed as part of the RI/FS site investigation. The operating procedures for the field analytical instruments will be followed and documented in the field notebook. Standard operating procedures (SOPs) for these analyses have been prepared and are included in Appendices A and B of the FSP.

8.1.1 Decontamination Procedures

All environmental sampling equipment will be decontaminated between sampling collection and again between sample locations. Rigorous decontamination procedures for heavy equipment are described in SOP 2 and for sampling equipment are described in SOP 3 (FSP).

8.1.2 Soil Borings

During the first phase of sampling activities, 39 soil borings (including temporary monitoring wells) will be advanced by direct-push technologies (e.g. Geoprobe^{TM}) to approximate depths of 15 feet below grade, top of the groundwater interface, or bedrock (whichever is achieved first). Five soil borings will be placed in each AOC (the center, the north, south, east, and west) to define the extent and magnitude of chemicals of concern in the site soil (Figure 3-1). The soil boring, located in the center of each area of concern, will be completed as a one-inch temporary monitoring well. These temporary monitoring wells will be utilized to determine the groundwater quality in each AOC. Soil samples will be collected continuously from the surface grade to the bottom of the boring in two-foot intervals. All soil samples will be field screened for VOCs, using a PID. A maximum of three grab soil samples from each boring will be submitted to the laboratory for analysis: a

surface soil sample with the highest PID reading from the depth interval of 0 to 3 feet bgs; a sample from the depth interval just above the groundwater interface; and the subsurface sample collected at the depth interval with the highest PID reading above the groundwater interface. If the sample from the depth interval just above the groundwater interface is also the sample with the highest PID reading, only two soil samples will be submitted for laboratory analysis from that boring. All soil samples submitted for laboratory analysis will be analyzed for VOCs, SVOCs, PCBs, and TAL metals (Table 7-1).

Soil samples will be analyzed for VOCs SW-846 Method 5035 (Low Level Scan) and will be preserved in the field following Methodology for Method 5035. The sample container for VOC analyses will come pre-preserved with sodium bisulfate (two VOA vials) and methanol (one VOA vial). Five gram of soil will be added to the preserved containers, which will then be placed on ice in a cooler.

Geotechnical samples will be collected (2 in natural soils and 2 in fill materials) after all soil borings have been completed to aid in the geotechnical sampling location placement. Geotechnical samples will be analyzed for grain size, moisture content, and atterberg limits. Results will be utilized in determining the screen slot size and sand pack to be used in monitoring well construction. In addition, geotechnical information will be utilized in site characterization.

8.1.3 Monitoring Well Construction

During the second phase of sampling activities, eight two-inch permanent monitoring wells will be installed at the site, five of which will be shallow (15 to 25 feet bgs) and three will be deep (40 to 50 feet bgs) to define the horizontal extent and magnitude of chemicals of concern in site groundwater. The locations of the permanent monitoring wells may be adjusted based on the preliminary results of the first phase of sampling activities. Soil borings, to be completed as permanent monitoring wells, will be advanced using a drill rig equipped with hollow stem augers. Soil samples will be collected continuously from the surface to the bottom of the boring in two-foot intervals. All samples will be field screened for VOCs, using a PID. A maximum of two grab soil samples will be submitted from each monitoring well location for laboratory analysis: the sample from the depth interval just above the groundwater interface and the subsurface sample with the highest PID reading

above the groundwater interface. If the sample from the depth interval just above the groundwater interface is also the sample with the highest PID reading, only one soil sample from that boring will be submitted for laboratory analysis.

Monitoring well sample locations will be placed in the field using aerial photographs. Following site investigation activities, monitoring wells will be professionally surveyed.

8.1.4 Monitoring Well Development

All new wells installed (temporary and permanent) and existing wells, as a part of this investigation, will be properly developed following procedures outlined in SOP 6 (FSP).

8.1.5 Groundwater Sampling

Groundwater samples and field measurements will be taken as a part of this site investigation. Groundwater sampling, using low-flow sampling techniques, and field measurement procedures will be performed in accordance with procedures outlined in SOP 7 (FSP).

8.1.6 Surface Water Sampling

To determine if surface water is impacted five surface water samples (including one to be collected directly from the storm sewer outfall pipe effluent that drains Locust Street and adjacent industrial facilities) will be collected and analyzed for VOCs, SVOCs, and TAL metals, based on preliminary results of the first phase of sampling activities. Samples will be analyzed as detailed in Table 7-1. The farthest downstream sample will be collected first, working to the farthest upstream sample to minimize suspended sediments from upstream impacting the downstream samples. In addition, the storm sewer will be surveyed with a camera to determine if any areas of the pipe have been damaged. Surface water sampling procedures will be performed in accordance with the procedures outline in SOP 13 (FSP).

8.1.7 Sediment Sampling

To determine if stream sediment has been impacted by former site operations, five sediment samples will be collected from identical locations as the surface water samples and analyzed for VOCs, SVOCs and metals, based on preliminary results from the first phase of sampling. Samples will be analyzed as detailed in Table 7-1. Surface water samples will be collected prior to the collection of the sediment samples. In addition, the farthest downstream sample will be collected first, working to the farthest upstream sample to

minimize suspended sediments from upstream impacting the downstream samples. Sediment samples will be collected following procedures outline in SOP 13 (FSP).

8.2 FIELD EQUIPMENT AND MATERIALS

Field equipment and materials needed to complete the activities to at the site, as outlined above, are itemized in the SOPs. Field sampling SOPs are located in Appendix A and B of the FSP. The following is a list of equipment and materials that will be utilized during the site investigation.

- *Shovels, picks, hand tools;*
- *Potable water;*
- *Steam cleaner/pressure washers;*
- *Decontamination pad;*
- *Phosphate-free detergent (e.g., Alconox or Liquinox);*
- *Analyte free water;*
- *Aluminum foil;*
- *Plastic/polyethylene sheeting;*
- *Plastic buckets and brushes;*
- *Field book and project plans;*
- *Metal detector;*
- *Marker stakes, flagging, and paint;*
- *Soil sample containers;*
- *Tape measure;*
- *Water level indicator;*
- *PID;*
- *Tape;*
- *Sample bottles;*
- *Coolers and ice;*
- *Shipping supplies;*
- *Waterra pump or Centrifugal pump;*
- *Generator and extension cords;*
- *In-line multi-parameter water quality meter;*
- *Stainless steel beaker (or similar);*
- *Low flow (less than 0.1 – 0.5 L/min) pump (bladder or electrical submersible); and*
- *Teflon tubing (dedicated).*

8.3 SUPPORT FACILITIES

No support facilities are needed for the collection of samples at the site. Prior to the initiation of fieldwork, a decontamination pad and area will be established. Decontamination of all heavy equipment will occur in this area. A Sample Packing Point will also be established, prior to site mobilization. All packing of samples will occur in this area.

8.4 HOLDING TIMES, PRESERVATION, AND TEMPERATURE

Table 8-1 lists the required containers, preservation, and holding times for each method. If holding times are exceeded, the laboratory is to contact the Project Manager and discuss whether samples should be analyzed outside of the holding time. Sample results are considered to be biased low as the time increases over the required holding time. When samples arrive at the laboratory, if temperatures are above the required limit, if preservative has not been added as required, bottles are leaking or there are bubbles in volatile vials, the laboratory is to contact the PM, immediately. The Parsons PM and the Laboratory Senior PM will determine if re-sampling is required.

8.5 INVESTIGATIVE DERIVED WASTE MANAGEMENT

Following completion of work, the site will be restored as close as possible to its pre-investigation condition. Drilling and sampling equipment will be removed upon completion of the drilling work. Drums will be moved to a centralized location until manifests can be obtained for disposal.

After each boring/well is completed, auger cuttings will be staged in 55-gallon Department of Transportation (D.O.T.) approved drums for subsequent handling. After the analytical results are received from the laboratory and validated, the final disposition of the soil will be determined. The contents of the drums will be analyzed for disposal criteria. These analyses include TCLP list, PCBs, corrosivity, reactivity, and ignitablity. After the final disposal of this waste is determined, Parsons will provide the logistical support for transportation and disposal activities. Soil samples not submitted for chemical analysis will be discarded. Discarded soil samples will be emptied from the sample containers and placed with the auger cuttings.

Liquids generated from decontamination (of heavy equipment and sampling equipment), well development, and purging will be stored in 55-gallon D.O.T. approved drums. A composite sample of the drums will be used to determine the characteristics of the collected water. If the water chemistry does not exceed method detection limits and/or background, it will be discharged to the ground. If the water is determined to be contaminated, additional characterization will be necessary and the materials will be disposed of accordingly.

IDW will be disposed, as required by Federal, state and local regulations, following methods outline in SOP 15 (Appendix A of the FSP).

8.6 FIELD PERFORMANCE CRITERIA

A summary of the performance criteria for each instrument that will be used to collect field measurements during sampling events is found in Table 8-2.

8.7 FIELD MEASUREMENT FAILURE

Samples will be collected and analyzed in the field during the scope of the RI/FS Site Investigation associated with this Quality Assurance Project Plan (QAPP). The internal quality control checks for these field measurements will include calibrating the instruments and are discussed in the SOPs applicable to the field measurement. These SOPs are included in the FSP. All data obtained in the field will be properly record in the field log book. Collection of the samples will be in accordance with the applicable procedures in the FSP. A list of the field Analytical Standard Operating Procedures that are included in the Appendix of the FSP and are listed in Table 8-3.

8.8 CORRECTIVE ACTION

Corrective action in the field can be needed when the sample network is changed (i.e. more/less samples, sampling locations, other than those specified in the work plan, etc.), or if sampling procedures require modifications due to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical or QA non-conformances or suspected deficiencies of any activity or issued document by reporting the situation to the RPM or designee. The Parsons PM will be responsible for assessing the suspected problems in consultation with the RPM and making a decision, based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable non-conformance requiring corrective action, then a non-conformance report will be initiated by the Project Manager.

The Project Manager will be responsible for ensuring that corrective action for non-conformances are initiated by:

- *Evaluating all reported non-conformances;*
- *Controlling additional work on non-conforming items;*
- *Determining disposition or action to be taken;*

- *Maintaining a log of non-conformances;*
- *Reviewing non-conformance reports and corrective actions taken; and*
- *Ensuring non-conformance reports are included in the final site documentation in project files.*

If appropriate, the Program Manager will ensure that no additional work that is dependent on the non-conforming activity is performed until the corrective actions are completed. The Field Team Leader or his designee is responsible for all site activities. In this role, the Data Acquisition Manager at times is required to adjust the site programs to accommodate site specific needs. When it becomes necessary to modify a program, the Data Acquisition Manager will notify the Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the RPM.

The change in the program will be documented on the field change request (FCR) that will be signed by the initiators and the Field Team Leader. The FCR for each document will be numbered serially, as required. The FCR will be attached to the file copy of the affected document. The RPM must approve the change in writing or verbally, prior to field implementation. The period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken.

Corrective action, resulting from internal field audits, will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Parsons Quality Assurance Officer will identify deficiencies and recommended corrective action to the Data Acquisition Manager. Implementation of corrective actions will be performed by the Data Acquisition Manager and Field Team Leader. Corrective action will be documented to the entire project management.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels.

Table 8-1
Sample Preservation, Handling, and Holding Times
CRS Site
Elyria, OH

Analysis	Method	Matrix	Sample Container	Preservation	Holding Time
VOCs	8260	Groundwater/ Surface Water	3 - 40 ml glass VOA vial with Teflon-lined septa and zero headspace	HCl	14 days
VOCs	5035	Soil/Sediment	3 - 40 ml glass VOA vial (2 with sodium bisulfate and 1 with methanol)	NA	7 days
SVOCs	8270	Groundwater/ Surface Water	1 liter amber glass jar with Teflon-lined lid	NA	7 days to extraction and 40 days after extraction
SVOCs	8270	Soil/ Sediment	8 or 16 oz wide mouth glass jar	NA	7 days to extraction and 40 days after extraction
PCBs	8082	Groundwater	1 liter amber glass jar with Teflon-lined lid	NA	7 days to extraction and 40 days after extraction
PCBs	8082	Soil	4 or 8 oz wide mouth glass jar	NA	7 days to extraction and 40 days after extraction

Table 8-1 (Continued)
Sample Preservation, Handling, and Holding Times
CRS Site
Elyria, OH

Analysis	Method	Matrix	Sample Container	Preservation	Holding Time
Metals	6020/6010 and 7470	Groundwater/ Surface Water	1 liter glass or polyethylene bottle	HNO_3	6 months
Metals	6020/6010 and 7470	Soil/ Sediment	8 or 16 oz wide mouth glass jar	NA	6 months
Methane, Ethane, and Ethene	RSK-175 Modified	Groundwater	3 - 40 ml glass VOA vial with Teflon-lined septa and zero headspace	HCl	7 days
Chloride	300.0A	Groundwater	250 ml plastic or glass jar	NA	28 days
Total Organic Carbon	415.1	Soil	NA	NA	NA
Moisture Content, Density of Soil, Grain Size Analysis	ASTM D2216, D2937, and D422	Soil	2-foot Shelby Tube sealed with wax.	NA	No Holding Time Requirements

Figure 9-1
Sample Labels
CRS Site
Elyria, OH

Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260
Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082
Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082	Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270
Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270	Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (filtered)
Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (unfiltered)	Site: CRS ID: **CRS-GW-MW-01** Date: Time: Analysis:

Figure 9-1 (Continued)
Sample Labels for Duplicate Samples
CRS Site
Elyria, OH

Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: VOCS SW-846 8260
Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: PCB SW-846 8082
Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: PCB SW-846 8082	Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: SVOCS SW-846 8270
Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: SVOCS SW-846 8270	Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (filtered)
Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (unfiltered)	Site: CRS ID: **CRS-FD-GW-DUP-A** Date: Time: Analysis:

Figure 9-1 (Continued)
Sample Labels for Matrix Spike (MS) Samples
CRS Site
Elyria, OH

Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260
Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082
Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082	Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270
Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270	Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (filtered)
Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (unfiltered)	Site: CRS ID: **CRS-MS-GW-MW-01** Date: Time: Analysis:

Figure 9-1 (Continued)
Sample Labels for Matrix Spike Duplicate (MSD) Samples
CRS Site
Elyria, OH

Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260
Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: VOCS SW-846 8260	Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082
Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: PCB SW-846 8082	Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270
Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: SVOCS SW-846 8270	Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (filtered)
Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis: Metals SW-846 6020/6010/7470 (unfiltered)	Site: CRS ID: **CRS-MSD-GW-MW-01** Date: Time: Analysis:

10.0 ANALYTICAL METHODS

The purpose of this Section is to identify the analytical methods to be followed, and how good the methods have to be, i.e., their performance criteria, to support any decisions to be made with the data.

10.1 LABORATORY

All samples (soil, groundwater, surface water, and sediment) collected during the field sampling activities for laboratory analysis will be analyzed by Severn Trent Laboratories, Inc. (STL), 4101 Shuffel Drive NW, North Canton, Ohio 44720, Telephone: (330)-497-9396.

10.1.1 Analytical Methods and Equipment Required

Samples at the site will be analyzed for several parameters, including both field and laboratory analysis (Table 7-1). All laboratory methods are USEPA SW 846 Methods, using currently promulgated revisions or Methods of Chemical Analysis of Water and Wastes (MCAWW). The following is a summary of each analyte group and laboratory method:

Analyte	Laboratory Method	Laboratory Equipment
Volatile Organic Compounds	SW-846 Method 8260 (groundwater/surface water)	GC/MS Tune
Volatile Organic Compounds	SW-846 Method 5035 (soil/sediment)	GC/MS Tune
Volatile Organic Compounds Prep/Volatile	Method 5030/35 (purge/trap) / 8260B (GCMS)	Purge/trap sampler
Semi Volatile Organic Compounds	SW-846 Method 8270C	GC/MS Tune
Semi Volatile Organic Compounds	Method 3520(w) 3540 (s) Method 8270 C (GCMS)	Purge/trap sampler
PCBs	SW-846 Method 8082	GC
Metals	SW-846 Method 6020/6010/7470	ICP / GFAA / MS
Mercury	Method 7470/71 (CVAA), includes prep	CVAA
Cyanide	MCAWW 335.3	Traacs 800
Metals Prep/Metals	Method 3005(w)/3050(s)/ 6010B (ICP or ICP Trace)	ICP

Table 10-1
STL, Inc. Standard Operating Procedures
CRS Site
Elyria, OH

SOP DESCRIPTION	METHOD REFERENCE	STL SOP NO.
Determination of VOCs by GC/MS based on Methods 8260B, 8260A, and 624	8260B (water)	CORP-MS-0002NC
Determination of VOCs by GC/MS based on Methods 8260B, 8260A, and 624 (Section 8 and 11)	5035 (soil/sediment)	CORP-MS-0002NC
GC/MS Analysis based on Methods 8270 C and 625 – Revision 2.4	8270C (water and soils)	CORP-MS-0001NC
GC Analysis based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8141A, 8015B, 608, 610, and Wisconsin DNR Modified DRO Method - Revision 5.5	8082 (water and soils)	CORP-GC-0001NC
Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, SW-846 Method 6010B and EPA Method 200.7	6010B (water and soils)	CORP-MT-0001NC
Inductively Couples Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8 (Supersedes 2.0)	6020 (water and soils)	NC-MT-0002
Preparation and analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW 846 7470 A and MCAWW 245.1 – Revision 2.2	7470A (waters)	CORP-MT-0005NC
Preparation and analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption, SW 846 7470 A and MCAWW 245.1 – Revision 2.2	7470 A (soils)	CP-MT-0007NC
Determination of Inorganic Anions by Ion Chromotography	Chloride 300.0A (waters)	NC-WC-0084
Analysis of Dissolved Gases in Groundwater by Modified Method RSK-175	RSK-175 Modified (waters)	NC-GC-0032
Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)	TOC 415.1 (soils)	NC-WC-0017
ASTM D2216 SOP for Water (Moisture) Content of Soil and Rock	ASTM D2216 (soil)	LM-SL-D2216
ASTM D2937 SOP for Measurement of Density of Soil in Place by the Drive Cylinder Method	ASTM D2937 (soil)	LM-SL-D2937
ASTM D422 SOP for Particle Size Analysis of Soils	ASTM D422 (soil)	LM-SL-D422
TBD – is being performed according to ASTM methodology	ASTM D5084 (soil)	TBD

Table 11-1 (Continued)
Data Quality Objectives and QC Criteria
CRS Site
Elyria, OH

QC Item	QC Criteria
I. PCB SW-846 Method 8082	
Initial Calibration (IC) (5 points), performed initially and when continuing calibration cannot be met. Includes a point at or near the reporting limit.	%RSD < 20%; or use a linear curve with $r \geq 0.995$.
Continuing Calibration (CC) Verification – once every 12 hours after the tune is verified but before samples are analyzed.	%Drift < 25% for target analytes
CC blank (instrument blank,) – once every 12 hours	< Reporting Limit
Preparation (method) blank, - one per QC batch	< Reporting Limit
MS/MSD, 1/20 samples or per SDG	Approved lab limits
Laboratory control sample 1/20 samples or per SDG	Approved lab limits
Surrogate TMX, DCB`	Approved lab limit
Field duplicate	< 50% RPD Results can be evaluated for precision, but this criteria will be considered as advisory only as the results are highly dependant on sample homogeneity and sampling technique.
DDT/ endrin break down	< 15%
Retention times	0.5 min of expected RT or ± 0.06 RRT units

Table 11-2
Method Specific Performance Criteria
CRS Site
Elyria, OH

Matrix Spike Recovery and Relative Percent Difference (RPD) Limits				
Parameter	**% Recovery**		**Water Matrix**	
GC/MS 8260B/8270C	**Water**	**Soil/Sediment**	**Water**	**Soil/Sediment**
1,1-Dichloroethylene	57-138	43-147	15	27
Trichloroethylene	58-141	46-143	17	23
Chlorobenzene	70-122	49-139	14	22
Toluene	67-129	46-147	14	24
Benzene	73-123	55-138	11	20
1,2,4-Trichlorobenzene	22-110	16-121	37	54
Acenaphthene	26-118	13-133	35	44
2,4-Dinitrotoluene	31-131	10-171	32	45
Pyrene	27-138	10-218	31	66
N-Nitroso-di-n-propylamine	18-115	12-128	36	50
1,4-Dichlorobenzene	18-110	18-110	36	59
Pentachlorophenol	10-140	10-144	56	87
Phenol	10-131	10-148	43	50
2-Chlorophenol	19-124	17-116	43	50
4-Chloro-3-methylphenol	21-124	17-128	55	55
4-Nitrophenol	10-145	10-148	34	64

Table 13-1
Laboratory Instrument Calibration
CRS Site
Elyria, OH

Analysis	Method Reference	Initial	Continuing	Ending
Chloride	300.0	5 levels plus blank "r" less than or equal to 0.995	1 level every 10 samples ± 10% of true value	1 level every 10 samples ± 10% of true value
Cyanide	335.3	7 levels plus blank "r" less than or equal to 0.995	1 mid level every 10 samples ± 10% of true value	1 mid level every 10 samples ± 10% of true value
TOC	415.1	3 levels plus blank	1 mid level every 10 samples ± 15% of true value	± 15% of true value
Metals	7000 series	3 levels plus blank ICV ± 10% of true value "r" less than or equal to 0.995	Every 10 samples ± 20% of true value	± 20% of true value
Metals	6020	3 levels plus blank ICV ± 10% of true value "r" less than or equal to 0.995	1 every 10 samples ± 10% of true value	± 5% of true value
Metals	6010	1 level and blank Rerun high calibration standard : verify quantitation at ± of true value, ICV< 5% from replicate	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD <5% replicate	Mid-level calibration standard ± 10% of true value CCV RSD <5% replicate
Mercury	7470/7470A	5 levels plus blank ICV ± 10% of true value "r" greater than or equal to 0.995	Every 10 sample ± 20% of true value	± 20% of original standard

Table 13-1 (Continued)
Laboratory Instrument Calibration
CRS Site
Elyria, OH

Analysis	Method Reference	Initial	Continuing	Ending
PCBs	8082	Minimum of 5 levels If % RSD <20%, use average RF. Otherwise, calibration curve employed. (See SOP # CORP-GC-0001)	Mid-level calibration standard analyzed every 10 samples. %D < 15% of predicted response for any analyte quantitiated and reported.	Mid-level calibration standard. %D < 15% of predicted response for any analyte quantitiated and reported.
VOCs	8260B	Minimum 5 levels % RSD for RF for CCCs < 30% SPCCs RF ≥ 0.300 for chlorobenzene and 1,1,2,2-tetrachloroethane, chloromethane and 1,1 dichloroethane, and RF > 0.100 for bromoform	Mid-level standard every 12 hours (after tuning) % Drift for CCCs < 20% between RF from standard and average RF from initial. SPCCs RF ≥ 0.300 for chlorobenzene and 1,1,2,2-tetrachloroethane, chloromethane and 1,1 dichloroethane, and RF > 0.100 for bromoform	NA
SVOCs	8270C	Minimum 5 levels % RSD for RF for CCCs < 30% SPCCs RF<0.050	Mid-level standard every 12 hours (after tuning) %D for CCCs <20% between RF from standard and average RF from initial SPCCs RF>0.050	NA

STL North Canton
STANDARD OPERATING PROCEDURE

TITLE: **INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY, EPA METHODS 6020 AND 200.8**

(SUPERSEDES: 2.0)

Prepared by: _____ 4-26-02
Date

Reviewed by: _____ 4.16.02
Technology Specialist Date

Approved by: _____ 4-16-02
Quality Assurance Manager Date

Approved by: _____ 4-22-02
Environmental Health and Safety Coordinator Date

Approved by: _____ 4-23-02
Laboratory Director Date

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on SW-846 protocol as described in EPA Method 6020 and 200.8. The source method lists fifteen elements approved for analysis by ICP/MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). Additional elements may be included provided that the method performance criteria presented in Section 9 is met. However, project approval may be required from the controlling agencies for compliance testing beyond the fifteen elements included in the promulgated method.

1.2. This procedure also describes the requirements for performing analysis of ground waters, surface waters and drinking water.

1.3. The procedure is applicable to the analysis of waters, soils, and wastes. No digestion is required prior to analysis for dissolved elements in water samples, but the samples must be filtered and preserved prior to analysis. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOP # CORP-IP-0002NC and SOP #CORP-IP-0003NC for preparation details.

1.4. The associated QuantIMs method codes are MH (6020) and QV (200.8).

1.5. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into an R.F. plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a resolution better than or equal to 0.9 AMU peak width at 10% of the peak height. For analysis by methods 200.8 the resolution requirement is 1.0 amu at 5% peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier. Interference must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and the constituents of the sample matrix. Recommended elemental equations which correct for many of these interferences are listed in Table I. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. *Dissolved Metals* - Those elements which pass through a 0.45 μm membrane filter (sample is acidified after filtration).

3.3. *Suspended Metals* - Those elements which are retained by a 0.45 μm membrane filter.

3.4. *Total Metals* - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5. *Total Recoverable Metals* - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6. *Instrument Detection Limit (IDL)* - See Section 9.1.1.

3.7. *Sensitivity* - The slope of the analytical curve (i.e. the functional relationship between raw instrument signal and the concentration).

3.8. *Tuning Solution* - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9. *Initial Calibration Verification/Quality Control Standard (ICV/QCS)* - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10. *Continuing Calibration Verification (CCV).* - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11. *Interference Check Standard (ICS)* - A solution containing both interfering and analyte elements of know concentration that is used to verify background and interelement correction factors.

3.12. *Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB)* - A multi-element standard of known concentrations which is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13. *Reagent Blank* - High purity (> 18 megohm-cm)water carried through the entire digestion process.

3.14. *Calibration Blank* - High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15. *Method Detection Limit (MDL).* See section 9.1.3

4. INTERFERENCES

4.1. Isobaric Interferences - Isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software.

4.2. Isobaric Molecular and Doubly Charged Ion Interferences - Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Table II lists isobaric interferences which might possibly affect required analytes. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.3. Physical Interferences - Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.3.1. Internal standards should be added at a level to give approximately 50,000 - 800,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 20 amu of the mass of the measured analyte.

4.3.2. Matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020 the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. If they fall outside this window, a five fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample reanalyzed. When performing method 200.8 the internal standards must be between 60% and 125% of the calibration blank. If they are outside this window the calibration blank is reanalyzed to verify internal standard intensities. If the intensities are within limits the sample is diluted by a factor of 2 (1:1) and reanalyzed.

4.3.3. Memory effects are dependent on the relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

4.3.4. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. Chemicals known to be **oxidizing agents** are: Nitric acid and hydrogen peroxide

5.3.2. The following materials are known to be **corrosive:** Sulfuric acid, hydrochloric acid, nitric acid, and hydrofluoric acid.

Note: Sulfuric and hydrochloric acids are used in cleaning the ICP/MS torch and hydrofluoric acid is also commonly used in air toxics preparations

5.4. The **RF Generator** produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and

prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Argon gas: High purity grade (99.99%).

6.2. Inductively Coupled Plasma Mass Spectrometer capable of providing resolution, less than or equal to 0.9 AMU at 10% peak height from 6-253 AMU and 1.0AMU at 5% peak height from 6-253 AMU with a data system that allows corrections for isobaric interferences and the application of the internal standard technique.

6.3. A four channel peristaltic pump.

6.4. Appropriate water cooling device.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Autosampler with autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Calibration standards are purchased as custom STL multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. See Table XI.

7.2. Check Calibration Standard (ICV)

A quality control standard similar to the calibration standards and prepared in the same acid matrix. This solution must be made at a concentration near the midpoint of the calibration curve. This standard is composed of analytes from a different source from those used in the calibration of the instrument. See Table XI.

7.3. The tuning solution is purchased as custom STL multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. The solution must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year.

7.4. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

7.5. Reagent water:

ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.6. Nitric acid, 5% HNO_3:

Carefully dilute 100 mL of concentrated HNO_3 to 2.0 L with reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of 2, and may be stored in plastic or glass. Preservation must be verified prior to analysis.

8.2. Soil samples do not require preservation, but must be stored at 4° ± 2°C until the time of preparation.

8.3. The analytical holding times for metals are six months from the time of collection.

8.4. Solid and aqueous samples must be digested prior to analysis by the appropriate method.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Table X provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. Prior to analysis of any analyte the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - IDLs can be determined by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as a separate analytical sample. The IDL should be performed every three months.

9.1.2. Linear Calibration Ranges - Linear calibration ranges are primarily detector limited. The linear range must be determined at instrument setup, and the upper limit must be verified annually or whenever a change in instrument hardware or operating conditions, in the judgement of the analyst, may lower expected ranges. Standards used to determine or verify linear ranges must be analyzed during a routine analytical run. The linear range is the concentration above which sample results cannot be reported.

9.1.3. For initial determination of the upper limit of the linear range, determine the signal responses from three different concentration standards across the estimated range. One standard must be at the upper limit of the estimated range. Results must recover within 10% of the expected value for the three standards. The Linear Range is then set at the concentration of the high standard.

9.1.4. For verification of the upper limit of the linear range, the high standard must recover within 10% of its expected value.

9.1.5. Method Detection Limit (MDL) - The laboratory shall determine a method detection limit for all analytes of interest initially and annually thereafter. The MDL study is performed and calculated according to STL Policy number CORP-QA-005, which is based on 40CFR Part 136 Appendix B.

9.2. Batch Definition

9.2.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.3. Method Blank

9.3.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead, calcium, magnesium, potassium, sodium or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.3.2. For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB

9.4. Laboratory Control Sample (LCS)

9.4.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The historical limits for the LCS for each analyte are in the LIMS system. Initial limits shall be set at 80-120% for method 6020 and 85-115% for method 200.8. If the LCS exceeds these limits for any analyte, that analyte is judged to be out of control and must be corrected before the analysis can be reported

9.4.2. Corrective Action for LCS

9.4.2.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.4.2.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4.2.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. The historical spike recovery acceptance limits for each analyte are in the LIMS

system. Initial limits shall be 70-130% for methods 6020 and 200.8. and the spike concentration must be the same level as the LFB. If they are not in control and all other quality control criteria have been met then a matrix interference is suspected.

9.5.2. Corrective action for MS/MSDs

9.5.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.5.2.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as NC (not calculated).

9.5.2.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the laboratory limits.

9.5.2.4. If client program requirements specify to confirm matrix interference's, repreparation and reanalysis of the MS/MSD may be necessary.

9.6. Sample Duplicate

9.6.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.6.2. Sample duplicates may be performed in lieu of or in addition to MSD's.

9.7. Control Limits

9.7.1. Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.7.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.8. Method Detection Limits (MDLs) and MDL Checks

9.8.1. MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021.

9.8.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.9. ICV/CCV/QCS - Calibration accuracy is verified at the beginning of each analytical run by analyzing a second-source initial calibration verification (ICV) standard. A continuing calibration verification (CCV) standard is analyzed at a 10% frequency throughout the run. The ICV must be within 10% of the expected value, or the analysis is terminated. The CCV must be within 10% of the expected value for method 6020 or 15% of the expected value for method 200.8, or the analysis is terminated and all samples since the last successful ICV/CCV reanalyzed.

9.10. RL Verification Standard –An independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. There is no set acceptance criteria established for this standard, but generally results should be within 50% of the expected value.

9.11. ICB/CCB/CB - The initial calibration blank must be analyzed immediately following the ICV. The continuing calibration blank must be analyzed at a frequency of 10% throughout the remainder of the analytical run. The ICB/CCB must be less than the reporting limit to be accepted.

9.12. Interference Check Solutions (ICSA/ICSAB) ***method 6020 only***- The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Table V. The interference check solutions must be analyzed at the beginning of every analytical run and every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences. See Table VI for analyte concentrations.

Note: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If

contamination can be confirmed by another method (ICP/GFAA), acceptance criteria will be applied at that level and the data accepted.

9.13. Internal Standards:

The intensities of all internal standards must be monitored throughout the run. The internal standard in the samples must be between 30% and 125% of the intensity of the calibration blank. If the sample falls outside of this criteria, perform the following procedures. First, evaluate nearby CCV's and CCB's. If sample internal standard recoveries appear to be related to instrument drift, then rerun affected samples. If sample internal standard recoveries appear to be primarily sample related, then perform serial fivefold (1+4) dilutions until the internal standard recoveries are within the 30-125% criteria. Alternately, the run may be reprocessed with an alternative internal standard that is not in the samples and at an appropriate mass for the masses being reported.

9.14. Serial Dilution ***method 6020 only*** - One serial five-fold dilution should be analyzed per batch for each matrix. If the analyte concentration is within linear range of the instrument and sufficiently high (generally, a factor of 100 times above the reporting limit), the serial dilution must agree within 10% of the original analysis. If not, an interference effect must be suspected, the result is flagged and included in the final report narrative. Samples identified as blanks cannot be used for serial dilution.

9.15. Post-Digestion Spike Addition (PDS) ***method 6020 only*** - If the serial dilution fails to meet the acceptance criteria, a PDS must be performed as follows. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered within 75 - 125% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected.

9.16. General Corrective Action Requirements - The general requirements for evaluation of QC results and corrective action for failures is described in STL Policy # QA-003

9.17. Nonconformance and Corrective Action

9.17.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument Start Up

Set up the instrument according to manufacturers operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning.

10.2. Instrument Tuning / Mass Calibration / Daily Performance

10.2.1. Daily Performance - Refer to the facility specific instrument SOP and ICP/MS instrument manual for detailed set up and operation protocols. Verify instrument performance daily with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 4 times. For method 200.8, the tuning solution must be analyzed 5 times with a relative standard deviation less than 5%.

10.2.2. Check mass calibration and resolution as needed.

10.2.2.1. Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

10.2.2.2. Mass Resolution Check - The resolution must be verified to be less than 0.9 amu full width at 10% peak height. Due to a limitation of the instrument software, the resolution requirement for method 200.8 of 1.0 amu full width at 5% peak height cannot be verified automatically. If the mass resolution requirement of 0.9AMU at 10% peak height is met the 200.8 requirement is also satisfied.

10.3. Calibrate the instrument for the analytes of interest according to manufacturer's instructions. Routine calibration and calibration verification levels are shown in Table XI. The calibration should include a blank and a three standards. For a linear, multi-point calibration curve, the correlation coefficient must be >/= 0.995. Report the average of at least three integrations for both calibration and sample analysis. A calibration must be performed daily and each time the instrument is set up.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOP # CORP-IP-0002NC and SOP #CORP-IP-0003NC for preparation details.

11.4. Sample Analysis

11.4.1. Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

11.4.2. Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element.

11.4.3. Dilute and reanalyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest . No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the IDL. (The sample should be diluted to the approximate midrange of the analytical curve.)

11.4.4. The analytical run sequence should be performed as follows to meet all quality control criteria:

Warm-up

Verify instrument performance

Calibration blank

Calibration standards

ICV

ICB

RL verification standard

ICSA (6020 Only)

ICSAB (6020 Only)

CCV

CCB

10 Samples

CCV

CCB

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100} \times \left(\frac{\mathbf{Found\ (ICV)}}{\mathbf{True\ (ICV)}} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100} \times \left(\frac{\mathbf{Found\ (CCV)}}{\mathbf{True\ (CCV)}} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \mathbf{100} \times \left(\frac{\mathbf{SSR - SR}}{\mathbf{SA}} \right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Note: When sample concentration is less than the method detection limit, use SR = 0 for purposes of calculating % Recovery.

12.4. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation:

$$RPD = \mathbf{100} \times \left[\frac{\mathbf{(DU1 - DU2)}}{\mathbf{(DU1 + DU2) / 2}} \right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$\text{Result (ug/L)} = \frac{\mathbf{(C \times V1 \times D)}}{\mathbf{V2}}$$

Where:
C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The concentration determined in digested solid samples when reported on a wet weight basis is as follows:

$$\text{Result (ug/kg)} = \frac{\mathbf{(C \times V \times D)}}{\mathbf{W}}$$

Where:
C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight, in g, of wet sample digested

12.7. Sample results should be reported according to the following significant figure rules:

12.7.1. All uncorrected values less than the detection limit are reported as "less than" the detection limit.

12.7.2. Positive results for target analytes are reported to three significant figures if the method is used without dilution.

12.8. Positive results obtained after dilution and results for non-certified analytes are reported to two significant figures

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Refer to Table 1 for the list of analytes that may be analyzed using this SOP for methods 6020 and 200.8.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within historical laboratory control limits and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.3. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualifications:

13.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Solvent waste must be disposed of in clearly labeled waste cans.

15.2. Acid waste must be collected in clearly labeled acid waste containers.

15.3. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.4. Refer to the Laboratory Sample and Waste Disposal plan.

15.5. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They

must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Test Methods For Evaluating Solid Waste, EPA SW-846, 3rd Edition, Final Update II, Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994.

16.1.2. Environmental Monitoring Systems Laboratory, EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry", Revision 5.4, EMMC Version

16.1.3. Corporate Quality Management Plan (QMP), current version.

16.1.4. STL Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Navy/Army SOP, NC-QA-0016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. Refer to Appendix for associated reporting limits

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method deviations

17.2.1. Deviations from method 6020

17.2.1.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.

17.2.1.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.2.1.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.

17.2.2. Deviations from method 200.8

17.2.2.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.

17.2.2.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.2.2.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18 megOhm) and by the analysis of blanks.

17.2.2.4. Resolution criteria of the mass calibration is met if the resolution criteria for method 6020 is satisfied.

17.2.2.5.The concentration of most analytes in the LCS is 100 µg/L. This is made from a commercially available stock solution and has all analytes at the same level. Verification records for this solution are on file with QA.

17.2.2.6. Internal standards are evaluated based upon a recovery criteria of 30% - 125%.

17.2.2.7. Results are reported up to the verified linear range, not up to only 90% of the linear range.

TABLE I: Recommended Elemental Interference Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

M = Total ion count rate at the specified mass

TABLE II: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS,SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

TABLE II: (cont.) Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Table III: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions**

	Molecular Interference	Nebulizer Flow Rate		
		High	Average	Low
Oxides:	ScO/Sc	0.00326	0.00055	0.00116
	YO/Y	0.00568	0.00395	0.00353
	TbO/Tb	0.0156	0.00648	0.00614
	ClO, Cl	0.00725	0.00227	0.00233
Hydroxides:	ScOH/Sc	0.00040	0.00011	0.00000
	YOH/Y	0.00078	0.00044	0.00048
	TbOH/Tb	0.00034	0.00008	0.00011
	ClOH/Cl	0.00048	0.00031	0.00029
Chlorine:	ClO/Cl	0.00725	0.00227	0.00233
	ClOH/Cl	0.00048	0.00031	0.00029
	ArCl/Cl	0.00605	0.00091	0.00477

** Information for this table is being determined by the EPA.

Table IV: Recommended Internal Standards

Method 6020	Method 200.8
Li	Sc
Sc	Y
Y	In
Rh	Tb
In	Bi
Tb	
Ho	
Bi	
Ge	

Table V: Interference Check Sample Components and Concentrations

(ICSAB minors are suggested spike levels)

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	50	50
Ca	50	50
Fe	50	50
Mg	50	50
Na	50	50
P	50	50
K	50	50
S	50	50
C	100	100
Cl	500	500
Mo	1.0	1.0
Ti	1.0	1.0
As	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Mn	0.0	0.1
Ni	0.0	0.1
Se	0.0	0.1
Ag	0.0	0.1
V	0.0	0.1
Zn	0.0	0.1

Table VI: Sample Preservation and Holding Times

Measurement Parameter	Container (1)	Preservative (2)	Maximum Holding Time (3)
Waters:			
Metals (4)	P,G	HNO_3 to pH < 2	6 months
Soils/Sediments/Wastes:			
The preservation required for soil/sediment/waste samples is maintenance at 4°C (± 2°C) until digestion.			

Footnotes:

(1) Polyethylene (P) or glass (G).

(2) Sample preservation is performed by the sampler immediately upon sample collection.

(3) Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that sample may be held before analysis and still considered valid. Holding times are calculated from the date when the sample was collected.

(4) Samples are filtered immediately on-site by the sampler before adding preservative for dissolved elements.

Table VII: Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in "quotes".

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"**205**", 203	Thallium
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
"98", 96, 92, **97**, 94	Molybdenum

Note: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Table VIII: Tuning Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below are two groups of suggested solutions which cover a typical mass calibration range.

Method 6020

Element	Concentration (µg/L)
Solution A	
Mg	10.
Rh	10.
Pb	10.
Solution B	
Li	10.
Co	10.
In	10.
Tl	10.

Method 200.8

Element	Concentration (µg/L)
Be	10.
Mg	10.
Co	10.
In	10.
Pb	10.

Table IX: Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>20,000
Rh	>200,000
Pb	>100,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

Table X: Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
ICV/QCS	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result must be < RL.	Terminate analysis; correct the problem; recalibrate.
CCV	Beginning and end of run and every 10 samples.	**6020**- 90 - 110% recovery. **200.8**- 85-115% recovery.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCB	Immediately following each CCV.	The result must be < RL.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA (6020 Only)	Beginning and every 12 hours.	Monitor for possible interferences.	See Section 9.12
ICSAB (6020 Only)	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.12

Table X (cont.) Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
Method Blank/Laboratory Reagent Blank	One per lot of 20 field samples or fewer.	The result must be < RL. Sample results greater than 20x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
Laboratory Control Sample/Laboratory Fortified Blank	One per lot of 20 field samples or fewer.	80-120%, or in-house limits (6020), 85-115% (200.8)	Redigest and reanalyze samples. See Section 9.4.
Serial Dilution (6020 Only)	One per lot of 20 field samples or fewer.	90 – 110% recovery	See section 9.14 for additional requirements.
Post-Digestion Spike (6020 Only)	See section 9.15	75-125% recovery	See section 9.15.
Matrix Spike/Matrix Spike Duplicate	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See section 9.5 for additional requirements.

Table XI: ICP/MS Calibration and Calibration Verification Checklist

Suggested Levels in µg/L

Element	Calibration			ICV	CCV
	1	2	3		
Aluminum	**10**	**50**	**100**	25	50
Antimony	10	50	100	25	50
Arsenic	10	50	100	25	50
Barium	10	50	100	25	50
Beryllium	10	50	100	25	50
Cadmium	10	50	100	25	50
Chromium	10	50	100	25	50
Cobalt	10	50	100	25	50
Copper	10	50	100	25	50
Lead	10	50	100	25	50
Manganese	10	50	100	25	50
Nickel	10	50	100	25	50
Silver	10	50	100	25	50
Thallium	10	50	100	25	50
Zinc	10	50	100	25	50

This procedure has been developed for additional elements. Additional elements may be included in the calibration solution at the appropriate levels. Levels may be adjusted to meet specific regulatory or client programs.

Table XII: Suggested ICP/MS Reporting Limits

Water 6020 and 200.8

Solid 6020 (only)

#	Compound	RL	Units	RL	Units
128	Antimony	2	ug/L	200	ug/kg
140	Arsenic	5	ug/L	500	ug/kg
194	Barium	5	ug/L	500	ug/kg
222	Beryllium	1	ug/L	100	ug/kg
411	Cadmium	1	ug/L	100	ug/kg
2952	Chromium	2	ug/L	200	ug/kg
637	Cobalt	1	ug/L	100	ug/kg
643	Copper	5	ug/L	500	ug/kg
1605	Lead	1	ug/L	100	ug/kg
1659	Manganese	5	ug/L	500	ug/kg
1906	Molybdenum	2	ug/L	200	ug/kg
1956	Nickel	2	ug/L	100	ug/kg
2281	Selenium	5	ug/L	500	ug/kg
2285	Silver	1	ug/L	100	ug/kg
2477	Thallium	1	ug/L	100	ug/kg
2479	Tin	10	ug/L	1000	ug/kg
2607	Vanadium	5	ug/L	500	ug/kg
2649	Zinc	10	ug/L	1000	ug/kg

APPENDIX A: DATA REVIEW CHECKLIST

STD1/CRI/QCSTD3 STD_____ ICSA/QCSTD4 STD_____

STD2/CCV/QCSTD6 STD_____ ICSAB/QCSTD5 STD_____

STD3 STD_____ ICV/QCSTD1 STD_____

QCSTD2= ICB QCSTD7 = CCB

STL North Canton ICP/MS Data Review Checklist

Run/Project Information:

Run Date: _____ Analyst:_____ Instrument:_____

Prep Batches Run:_____

_____

Circle Methods used: 6020/ 200.8: CORP-MT-0001NC

Review Items

A. Tune/Daily Performance	Yes	No	N/A	2nd Level
1. Resolution </= 0.9 AMU full width at 10% peak height, and within +/- 0.1 AMU of true mass?				
2. Performance check within recommended specfications? (Mg>10,000cps) (Rh >75,000 cps) (Pb >50,000 cps) (CeO/Ce ≤ 0.03) (Ba++/Ba+ ≤ 0.03) (Background < 30cps @ Mass 220)				
B. Calibration/Instrument Run QC				
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ? Correlation coefficient ≥ .995 ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits? *(ICV:* = 90 – 110%) *(CCV: 90 – 110%,* 200.8 = 85 –115% *)*				

3. ICB/CCB analyzed at appropriate frequency and within +/- RL?				
4. CRI run and recovered within QC limits (+/-50%)?				
5. ICSA/ICSAB run at required frequency and within SOP control limits?				
C. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
D. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Serial dilution done per prep batch?				
6. Post digest spike analyzed if required?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Level I Analyst:_____ **Date:**_____ **Time:**_____

Comments:_____

Level II Reviewer:_____ **Date:**_____ **Time:**_____

Comments:_____

APPENDIX B – OPERATION INSTRUCTIONS – P.E. 6100

ICP/MS INSTRUCTIONS

A. Light the plasma and start the peristaltic pump.

1. Allow the instrument to warm up approximately 10-15 minutes.

B. Daily Performance

1. Open Daily2_asx workplace.
2. Open the sampling tab in the method screen.
3. Select and initialize the autosampler. Click O.K.
4. Click on probe. Click go to rinse. Send probe to tube #8.
5. Allow solution to reach plasma and then click on analyze sample.
6. When analysis is complete, send probe back to rinse.

C. Analysis Setup

1. Open Analysis workspace.
2. Under the method window, open the report tab and type in your report filename. Save the method.
3. Highlight the dataset window. Go to file and select new. Type in the dataset name (normally the same as the filename).
4. Highlight autosampler window. Type in your autosampler locations and samples. Right click under measurement action and select what needs to be analyzed. Right click under method description and select method. Verify that all times and rpms on table are correct. Go to file and click save as. Type autosampler table name (normally the same as the filename).

D. Analyze samples

1. Highlight samples to be analyzed.
2. Left click on analyze batch.

APPENDIX C – OPTIMIZATIONS – P.E. 6100

X-Y ADJUSTMENT – Adjusts torch to achieve best intensities. This should be done whenever anything is done to the torch or the cones.

1. Open X-Y_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow solution to reach plasma and hit analyze sample.
4. Adjust x and y while watching the signal on the realtime window. Adjust only until signal is at its highest.

NEB LENS – This also is done to get best intensities. It effects the shape and the depth of the plasma. This should be done if your oxides or doubly charged are >3%.

1. Open neb lens power oxides.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the Autooptimize tab. Select nebulizer gas flow.
4. Click get analyte list.
5. Make sure solution has reached the plasma and click optimize.
6. When done, save the optimization file.

AUTOLENS – Each element done represents a section of the mass spectrum. Be is on the low end, CO is in the middle and IN is on the higher end. If there is a problem with any particular section of the spectrum, it may be an indication that this needs done. This should probably be performed on a weekly basis.

1. Open Autolens_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the autolens tab.
4. Click on clear calibration.
5. Click on get analyte list.

6. Make sure the solution has reached the plasma and click on calibrate.
7. Save the optimization file.
8. In the interactive window, the optimization curve can be printed.

DUAL DETECTOR CALIBRATION – This extends the dynamic range of the detector. This should be done on a weekly basis. All analytes of interest must be included in the solution.

1. Open dual detector2.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on dual detector cal tab.
4. Click on clear calibration.
5. Click on get analyte list.
6. Make sure the solution has reached the plasma and click on calibrate.
7. When complete, check to make sure that all r values are at least .999.
8. Save the optimization file.
9. In the interactive window, the plots can be displayed.

TUNING – This adjusts the electronics to assure the accuracy of the mass spec. The resolution adjustment assures that the resolution of each mass of interest is within range. This should be done monthly.

1. Open tuning_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow sample to reach the plasma and click on tune mass spec.
4. Make sure measured mass and peak width is within range. If its not, resolution will need adjusted. Adding 30 units will decrease the amu by about 0.1. Subtracting 30 units will increase the amu about 0.1.

GENERAL NOTES –

1. All optimizations can be done with the tuning solution, except the dual detector cal needs the cross cal standard with all the elements of interest.
2. All optimizations can be run at 12 rpm, except the dual detector calibration should be done at 24 rpm.

Appendix D – ICPMS Maintenance Schedule

Daily	Change sample and internal standard pump tubing and pump windings
	Check argon gas supply level
	Check rinse solution and fill if needed
	Check waste containers and empty if needed
	Check sample capillary tubing is clean and in good condition
	Check sample flow for cross flow nebulizer
	Check pressure for vacuum systems
	Check daily performance
As Needed	Clean Sampler and skimmer cones
	Clean plasma torch assembly to remove accumulated deposits
	Clean nebulizer
	Replace sample and internal standard capillary tubing and autosampler sipper probe
	Replace drain tubing
	Perform necessary optimizations
	Clean autolens
Monthly	Inspect air filters; clean or replace as needed
Bi-Yearly	Change oil in vacuum pumps
	Check water in coolflow